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Flexural Strength and Physical Changes of Dental Composite Resins after Extract Treatments

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Recently introduced composite resins for anterior or posterior tooth may improve physical strength and polymerization. It is also becoming important that biocompatible or biodegradable materials are used. In order to get a Korean admission for making or selling any dental resin's material, the tests include flexural strength of composite resin and quantitative analysis after extract treatment. The purpose of this study was to test the effect of extract conditions such as extract time and temperature on the composite resins and then evaluate them by flexural strength, UV absorbance, and potassium permanganate consumption. Four commercial products, AELITE™ Universal Light-Cured (Bisco, Schaumberg, IL USA), GRADIA DIRECT ANTERIOR (GC Corp., Tokyo, Japan), DenFil™ (Vericom, Anyang, Korea), and Filtek™ Z350 (3M ESPE, St. Paul, MN USA), were used for this study. Four conditions of the extract treatment were (121 ± 2°C, 1 ± 0.2 hr), (70 ± 2°C, 24 ± 2 hr), (50 ± 2°C, 72 ± 2 hr), and (37 ± 2°C, 72 ± 2 hr), respectively. The cured samples were examined using a Universal Testing Machine (Instron 3366, Instron Engineering Corporation, Canton, MA USA). After treatment, the elution for the quantitative analysis was measured using a UV-vis spectrophotometer (WPA S2100, Biochrom Ltd., Cambridge, UK). To quantify chemically reduced materials, the test of potassium permanganate reduction material ($KMnO_4$-test) was required. From the present study, we concluded that the results indicated both extraction time and temperature affected the flexural strength, UV absorbance, and $KMnO_4$-test of the dental composite resins. Overall, increasing extraction time weakened the composite resins while increasing extraction temperature heightened the flexural strength of the composite resins. The UV absorbance was higher when the extract conditions were high temperature and short time duration then compared to low temperature and long time duration. In certain cases, $KMnO_4$-test showed the extract time was more important than the extract temperature. These are cautiously expected that both extract temperature and time influence flexural strength; extract temperature is more significant to UV absorbed materials and extract time is more important in terms of chemically reduced materials.

Key words: Composite resin, Extraction treatment, Flexural strength, UV Absorbance, Potassium permanganate consumption

Introduction

Dental resin-based composites generally consist of a matrix that is formed by a plastic resin material, fillers which are reinforcing particles and/or fibers dispersed in the matrix, and a coupling agent between filler and resin matrix. Matrix has two partitions such as oligomers like bisphenol A-glycidyl methacrylate (bis-GMA), bisphenol A-polyethylene glycol diether dimethacrylate (bis-EMA), or urethane dimethacrylate (UDMA) and viscosity controllers like methyl methacrylate (MMA), ethylenedimethacrylate (EDMA), or triethyleneglycol dimethacrylate (TEGDMA). The strength and the range of use depend on the ratio of resin materials and fillers. Light-activated resins also need a photosensitizer like camphorquinone (CQ) and an amine initiator and/or electron donor like dimethylaminoethyl methacrylate (DMAEMA). In addition to the dimetlyacrylates, other multifunctional resins have been introduced to dentistry as dentin bonding agents like dipentaerythiol penta-acrylate monophosphate (PENTA-P) or poly acrylic acid (PAA) which hydroxyethyl methacrylate (HEMA) has been grafted from.[1)]

Recently introduced composite resins for anterior or posterior tooth may improve physical strength and polymerization. As the use of biocompatible or biodegradable materials is becoming more important, the chemical degradation of methacylate monomers has been a subject of research to clarify the hydrolytic degradation behavior of ester linkages in polymer networks.[2-4)] Degradation of dental resins in human saliva, which lead to decrease in mechanical strength and surface hardness, have been also reported in *in vitro* studies.[5-8)]

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Table 1. General resin degradation processes

Physical Processes	Chemical Processes
A. Material loss or uptake	A. Thermolysis
Sorption	Scission reactions generating radicals
Swelling	Depolymerization
Softening	B. Oxidation
Dissolution	Chemical
Mineralization	Thermo-oxidative
Extraction	C. Solvolysis
B. Physical changes	Hydrolysis
Crystallization	Alcoholysis, etc.
Decrystallization	D. Photolysis
Stress cracking	E. Radiolysis
Fatigue fracture	
Impact fracture	

To get a Korean admission for making or selling any dental resin's material, the tests include ISO and Korean Pharmacopoeia.[10-12] ISO 4049 requires the tests like sensitivity to ambient light, depth of cure, flexural strength, water sorption and solubility, shade and color stability after irradiation and water sorption, and radio-opacity as light-activated composite resins. Since resin medical materials depend on the extract conditions, and Korean Pharmacopoeia requires the tests such as appearance, pH, heavy metal, potassium permanganate consumption ($KMnO_4$-test), residue after evaporation, and UV absorbance.

The purpose of this study was to test the effect of extract condition such as extract time and temperature on the composite resins and evaluate them by flexural strength, UV absorbance, and potassium permanganate consumption.

Materials and Methods

Five composite resin specimens which were 25 mm × 2 mm × 2 mm in dimension were light-cured for 20 seconds on each side as indicated by the respective manufacturers for each of the four composite resin types (classified *Class 2*, *Group 1* of *Type 2* by ISO 4049:2000(E)): AELITE™ Universal Light-Cured (Bisco, Schaumberg, IL USA (AELITE)), GRADIA DIRECT ANTERIOR (GC Corp., Tokyo, Japan (GRADIA)), Den-Fil™ (Vericom, Anyang, Korea (DenFil)), and Filtek™ Z350 (3M ESPE, St. Paul, MN USA (Filtek)). These resin specimens made up one temperature and time experimental group; (121 ± 2°C, 1 ± 0.2 hr), (70 ± 2°C, 24 ± 2 hr), (50 ± 2°C, (72 ± 2 hr), and (37 ± 2°C, 72 ± 2 hr), respectively. Control group was stored in distilled water at 37 ± 1°C for 24±2 hours, and experimental group resin specimens were stored in distilled water at four different temperatures and times. After treatment, loads were applied (cross-head speed of (0.75 ± 0.25) mm/min) using Universal Testing Machine (Instron 3366, Instron Engineering Corporation, Canton, MA USA) until breaking point and the results were statistically analyzed by the Friedman Test and Kruskal-Wallis Test ($p < 0.05$, $n = 5$).

Composite-resin specimens which were 15 ± 0.1 mm in diameter and 1.0 ± 0.1 mm in thickness were light-cured for 20 seconds on each side as indicated by the manufacturers for each of upper four composite resins. Each group of specimens was stored in distilled water (20 ml/4 g) at the four different temperatures and times. For UV-vis spectroscopy, absorbance was measured using a UV-vis spectrophotometer (WPA S2100, Biochrom Ltd., Cambridge, UK). The control

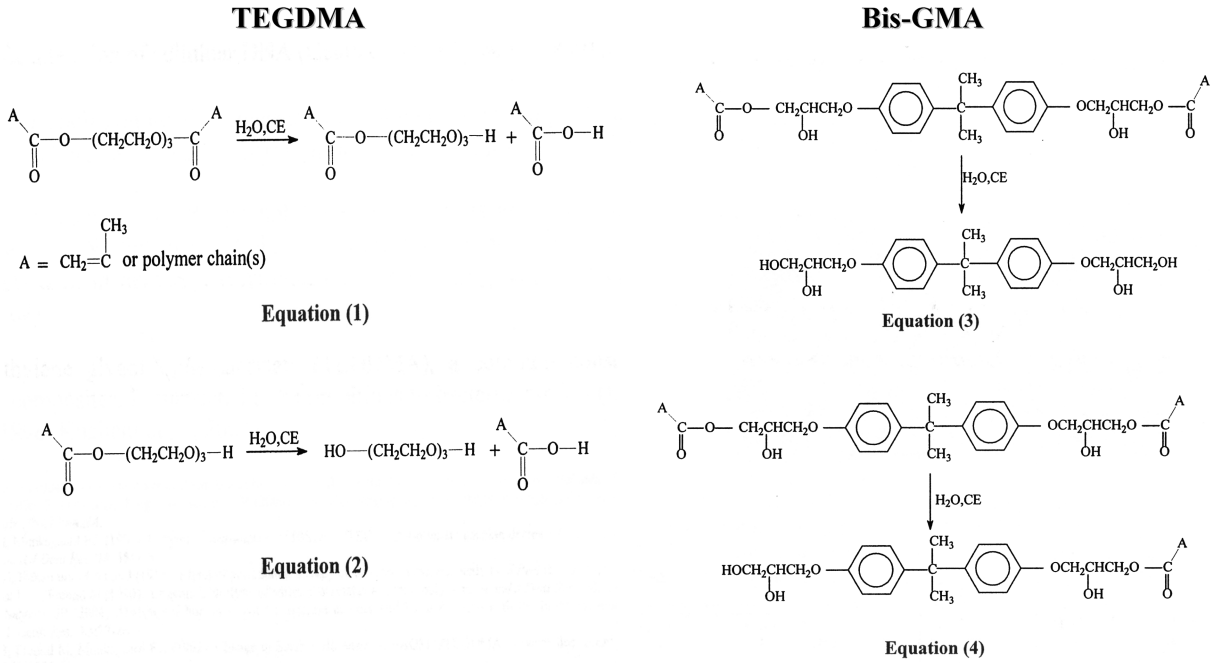

Figure 1. resin degradation products by hydrolysis[9]

consisted of distilled water instead of the eluted extract statistically analyzed by Friedman Test and Kruskal-Wallis Test *($p<0.05$, $n=5$)*.

For $KMnO_4$-test, 20 ml resin extract was added to 20 ml 0.002 M $KMnO_4$ and 1 ml dil. H_2SO_4. This mixture was boiled for 3 minutes, and KI was added. The mixture was then back-titrated with 0.01 M sodium thiosulfate. The differences of titrated solution of sodium thiosulfate between experimental and control groups were calculated. The control consisted of distilled water instead of the eluted extract statistically analyzed by Friedman Test and Kruskal-Wallis Test *($p>0.05$, $n=5$)*.

Results and Discussion

The results indicated that both extraction time and temperature affected the flexural strength of the dental composite resins (Table 2). Nevertheless, under the tested extraction conditions, all composite resins were able to withstand substantially more than 50 MPa as specified by ISO 4049. Overall, increasing extraction time weakened the composite resins while increasing extraction temperature heightened the flexural strength of the composite resins. Although the Filtek Z350 flexural strength did not increase significantly as temperature increased/decreased and as time increased, the Filtek Z350 resin composite consistently showed the greatest flexural strength except for the control. DenFil had the largest MPa value, but the difference was not significant. GRADIA composite resin also did not fluctuate much in flexural strength under different extraction conditions. However, even when the GRADIA composite provided near linear values, GRADIA consistently produced the lowest flexural strength values. The significant difference in flexural strength values among the composite resins is clearly evident in the control test group. Low flexural strength would be less-than-ideal in practice. Perhaps these low values can be explained by the fact that GRADIA was specifically made for anterior tooth surfaces where resin strength would have less emphasis than it would have on occlusal surfaces. Thus, Filtek Z350 seemed to offer the most consistent set of high flexural strength values. DenFil and AELITE produced the next highest set of strength values respectively, although there was greater time and temperature dependence for these two resins. As a whole, the Filtek Z350, DenFil, and AELITE resins produced significantly higher flexural strength values than GRADIA. This can be related to the fact that these three kinds of resins were made for both anterior and posterior tooth surfaces.

The results indicated that extraction temperature affected the UV absorbance of the dental composite resins (Table 3). Although the GRADIA UV absorbance did not increase significantly as temperature increased, there was a noticeable trend in increased absorbance as increased temperature. All absorbencies of extracts from GRADIA produced the lowest peaks at below 0.100 which was favorable. Since GRADIA was made for only anterior tooth surface, it would consist of more polymer than the other composite resins. Next to GRADIA, DenFil was extracted the smallest volume and AELITE was the largest volume among the three composite resins for both of

Table 2. Flexural strength of dental composite resins after extract treatments (MPa)

	Control Group	Experimental Groups			
		121 ± 2°C, 1 ± 0.2 hr	70 ± 2°C, 24 ± 2 hr	50 ± 2°C, 72 ± 2 hr	37 ± 2°C, 72 ± 2 hr
AELITE	110.22 (15.01)^{A,c}	102.91 (6.62)^{B,c}	95.62 (4.10)^{C,c}	88.58 (17.01)^{D,a}	77.83 (5.27)^{E,d}
GRADIA	83.36 (4.21)^{A,d}	95.41 (8.64)^{A,d}	86.73 (4.29)^{A,d}	86.00 (7.49)^{A,a}	85.76 (4.30)^{A,c}
DenFil	118.42 (7.57)^{A,a}	106.56 (2.85)^{B,b}	104.20 (6.80)^{C,b}	101.31 (5.56)^{D,a}	95.42 (9.11)^{E,b}
Filtek Z350	115.40 (12.63)^{A,b}	108.05 (2.73)^{A,a}	106.79 (6.98)^{A,a}	106.40 (2.49)^{A,a}	103.45 (9.36)^{A,a}

( ): Standard deviation.
A,B,C,D, and E: Significant difference in the row by Kruskal-Wallis Test *($p<0.05$)*.
a,b,c,d, and e: Significant difference in the column by Friedman Test *($p<0.05$)*.

Table 3. UV absorbance of dental composite resins after extract treatments

	121 ± 2°C, 1 ± 0.2 hr	70 ± 2°C, 24 ± 2 hr	50 ± 2°C, 72 ± 2 hr	37 ± 2°C, 72 ± 2 hr
AELITE	1.362 (0.64)^{A,a}	1.284 (0.59)^{B,a}	0.644 (0.05)^{C,a}	0.489 (0.02)^{D,a}
GRADIA	0.023 (0.01)^{A,d}	0.022 (0.01)^{A,d}	0.019 (0.01)^{A,d}	0.009 (0.01)^{A,d}
DenFil	0.277 (0.04)^{A,c}	0..268 (0.05)^{B,c}	0.233 (0.08)^{C,b}	0.131 (0.02)^{D,b}
Filtek Z350	0.677 (0.07)^{A,b}	0.669 (0.07)^{B,b}	0.182 (0.12)^{C,c}	0.129 (0.10)^{D,c}

( ): Standard deviation
A,B,C, and D: Significant difference in the row by Kruskal-Wallis Test *($p<0.05$)*
a,b,c, and d: Significant difference in the column by Friedman Test *($p<0.05$)*

Table 4. $KMnO_4$-test of dental composite resins after extract treatments (ml)

	121 ± 2°C, 1 ± 0.2 hr	70 ± 2°C, 24 ± 2 hr	50 ± 2°C, 72 ± 2 hr	37 ± 2°C, 72 ± 2 hr
AELITE	19.4 (1.64)^{A,a}	21.0 (0.99)^{A,a}	11.7 (0.45)^{C,a}	11.1 (1.02)^{D,a}
GRADIA	19.1 (1.59)^{A,a}	12.0 (0.12)^{A,d}	14.6 (0.55)^{A,d}	13.0 (0.48)^{A,d}
DenFil	15.8 (0.87)^{A,a}	13.2 (0.75)^{B,c}	11.6 (0.88)^{C,b}	16.0 (0.42)^{D,b}
Filtek Z350	12.6 (1.59)^{A,a}	12.8 (1.07)^{B,b}	17.2 (0.45)^{C,c}	12.0 (0.50)^{D,c}

( ): Standard deviation

anterior and posterior tooth surfaces. Extraction temperature was more important than extraction time. Because their compositions from each company were made of different resin matrix and fillers, there were probably distinctions between results.

The results indicated that extraction temperature and time affected the chemically reduced material of the dental composite resins (Table 4). The consumption of the control group was 20 to 23 ml. The larger the difference between the control and the experimental groups, the higher the value of $KMnO_4$-test's result. The extract condition of high temperature and short time duration usually showed higher difference than that of low temperature and long time duration. The extract time, in certain cases, was more important than the extract temperature because 70 ± 2°C, 24 ± 2 hr and 121 ± 2°C, 1 ± 0.2 hr produced variable results. From these reasons, there was no significantly difference between conditions.

Conclusions

Both extract temperature and time influenced composite resin's flexural strength, UV absorbance and $KMnO_4$-test. Increasing extraction time weakened the composite resins while increasing extraction temperature heightened the flexural strength of the composite resins. The extract condition of high temperature and short time duration showed higher UV absorbance than that of low temperature and long time. Sometimes, $KMnO_4$-test showed the extract time was more important than the extract temperature. These are cautiously expected that both extract temperature and time influence flexural strength; extract temperature is more significant to UV absorbed materials and extract time is more important in terms of chemically reduced materials.

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